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Patent #1734

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Serial No. 09/336,245

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For: DIE ATTACH ADHESIVES FOR USE IN  
MICROELECTRONIC DEVICES

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SIR:

*not an IDS, just translation  
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initial IDS.*

Please find enclosed the **translation** of the Japanese reference 10,168413 submitted in the Information Disclosure Statement mailed on **December 12, 2000**.

Respectfully submitted,

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Patent Application No.:	8-335782
Patent Application Date:	December 16, 1996
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Title of Invention:

Anisotropic conductive adhesive

Abstract:

Purpose of Invention: To provide a thermosetting anisotropic conductive adhesive with excellent adhesiveness, connection reliability, storage stability, and repairability, which is able to form electrical connections between fine circuits, such as LCDs and TCPs or TCPs and PCBs, especially at low temperatures and in short periods of time.

Make-up of Invention: An anisotropic conductive adhesive, characterized in that it contains, as necessary ingredients, a radical-polymerizable resin (A), a radical-polymerizable monomer (B), an organic peroxide (C), a thermoplastic elastomer (D), maleimide (E), and conductive particles dispersed in these resin compositions [*sic*], and the proportions of these ingredients, in parts by weight, are in the ranges of

$$(1) \quad (C)/\{(A)+(B)+(E)\}=(0.1-15)/100$$

$$(2) \quad (B)/(A)=(1-90)/100.$$

Claims:

(1) An anisotropic conductive adhesive, characterized in that it contains, as necessary ingredients, a radical-polymerizable resin (A), a radical-polymerizable monomer (B), an organic peroxide (C), a thermoplastic elastomer (D), maleimide (E), and conductive particles dispersed in these resin compositions, and the proportions of these ingredients, in parts by weight, are in the ranges of

$$(1) \quad (C)/\{(A)+(B)+(E)\}=(0.1-15)/100$$

$$(2) \quad (B)/(A)=(1-90)/100.$$

(2) An anisotropic conductive adhesive in accordance with Claim (1), characterized in that the radical-polymerizable resin is a vinyl ester resin.

(3) An anisotropic conductive adhesive in accordance with Claim (1), characterized in that the thermoplastic elastomer is a copolymer the principal ingredients of which are acrylonitrile and butadiene.

## Detailed Explanation of Invention:

### Industrial Field of Application

This invention concerns an anisotropic conductive adhesive which is used for electrically connecting fine circuits, such as LCDs (liquid crystal displays) and TCPs (tape carrier packages), or TCPs and PCBs (printer circuit boards), etc.

### Prior Art

In recent years, the necessity for using anisotropic conductive adhesives, containing conductive particles dispersed in adhesive resins, for connecting various kinds of fine circuits, such as liquid crystal displays (LCDs) and TCPs, or TCPs and PCBs, has increased dramatically, and such adhesives have been used as a method of making these connections. With this method, electrical insulating between adjacent terminals in the same plane is maintained, while current is allowed to flow between upper and lower terminals, by sandwiching the anisotropic conductive adhesive between the parts to be connected and applying heat and pressure. The reason why anisotropic conductive adhesives are frequently used in such applications is that conventional connection methods, such as soldering, cannot be employed because the parts which are to be adhered together are not heat-resistant, and adjacent terminals in fine circuits are shorted when the conventional methods are used. These anisotropic conductive adhesives can be classified into thermoplastic and thermosetting types; recently, the thermosetting epoxy resin type, which has excellent reliability, has been more widely used than the thermoplastic type.

Styrene copolymers, such as SBS (styrene-butadiene-styrene), SIS (styrene-isoprene-styrene), SEBS (styrene-ethylene-butadiene-styrene), etc., have been used for the most part as anisotropic conductive adhesives of the thermoplastic type. The method of using these thermoplastic anisotropic conductive adhesives is fundamentally that of melting and fusing them. It is thought that the workability of this method is generally good, since, if [proper] conditions are selected, the method can be applied at relatively low temperatures and with short periods of time, compared with the thermosetting type. However, since the moisture resistances, chemical resistances, etc., of these resins are low, the reliability of the connection is low, and these adhesives have not been able to stand up to long-term environmental tests.

On the other hand, the anisotropic conductive adhesives of the thermosetting type, which are presently the most often used ones, are generally epoxy resin thermosetting adhesives, which have a good balance between storage stability and thermosetting properties. However, in actual use, these thermosetting adhesives need to be set by heating them at 150–200°C for about 30 seconds, due to their setting reactivities, in order to obtain both the storage stability and the resin setting ability. Therefore, it has been difficult to set the resins at temperatures of 150°C or lower, for example, within practical adhesive times. Furthermore, for reasons of storage stability, compositions containing latent curing agents, such as BF<sub>3</sub> amine complexes, dicyandiamide, organic acid hydrazides, imidazole compounds, etc., have been proposed, but these compositions have both advantages and disadvantages: the ones which have excellent storage stability require long times or high temperatures to cure, whereas those which cure at low temperatures in short periods of time are inferior in storage stability.

In addition to these problems, in the operability of connecting fine circuits together using thermosetting anisotropic conductive adhesives, there is frequently a desire to be able to peel the connected parts apart, after they have been connected, and reconnect them (so-called "repairing"), without breaking or damaging them, due to incorrect positioning, etc. However, almost all of these adhesives, together with their advantages of high adhesive strength and high reliability, also have the drawback that it is very difficult for them to meet these seemingly contradictory demands, and satisfactory adhesives in this respect have not been obtained. Recently, in particular, there have been rapid advances in increasing the areas of LCD modules, as well as increasing their precision and forming narrower frames. As a consequence of this, the pitches of the connections have rapidly become finer and the widths of the connections have rapidly decreased. Therefore, in connections between LCDs and TCPs, for example, mismatches of the connecting patterns are produced because of elongation of the TCPs during the connecting operation, and the fine widths of the connecting parts have given rise to the problem of thermal effects on the parts in the LCDs at the temperatures used during the connection. Furthermore, when TCPs and PCBs are connected, the PCBs lengthen, so that the PCBs and LCDs are warped by the heat of the connecting operation and the TCP wiring breaks.

Therefore, it was thought that these problems could be solved by performing the connections at lower temperatures, but if one tries to make the connections with conventional thermoplastic anisotropic conductive adhesives, the problem arises that, although the connections can be made at relatively low temperatures, the reliability of the connections will be bad because of the low moisture and heat resistances of the resin. Moreover, if one tries to use epoxy resin anisotropic conductive adhesives, which are the main thermosetting type used, at low temperatures, the connection time must be made long in order to cure the resin, and these adhesives cannot be used in practical applications. As anisotropic conductive adhesives which can be used to make connections at low temperatures, an adhesive with conductive particles dispersed in an adhesive resin in which a cation-polymerizable substance and a sulfonium salt are compounded (Japan Public Patent Disclosure Bulletin No. 7-90237), and an adhesive in which the conductive particles are dispersed in an epoxy resin and a 4-(dialkylamino)pyridine derivative (Japan Public Patent Disclosure Bulletin No. 4-189883) have been proposed, but these adhesives have the problems with the storability of the adhesive resins, corrosion of the adhered circuit terminals, etc., so that they are not practical.

Furthermore, it has been thought that thermosetting anisotropic conductive adhesives with conductive particles dispersed in adhesives with excellent curing abilities at low temperatures and in short times, consisting of radical-polymerizable resins, organic peroxides, and thermoplastic elastomers, can be used to make possible low-temperature connections, but they have the problem that the radical-polymerizable resin and the thermoplastic elastomer separate at the time of the heating and adhesiveness, because of differences in compatibility, and sufficient adhesive strength and stability of connection cannot be achieved. As a method of solving this problem of separation during heating and adhering, the compounding of maleimides, which are compatible with both the radical-polymerizable resin and the thermoplastic elastomer, has been proposed. However, this method presents the problem that, besides the curing ability being reduced by the compounding of the maleimides, the melting points of the radical-polymerizable resin and the maleimide are not sufficiently low with respect to the temperature of the heating and adhering, so that the resin flowability at the time of the adhesion is insufficient,

which causes poor conductivity and increased connection resistance, or the cured adhesive becomes hard and brittle, reducing the adhesive force. Therefore, this method is not practical. That is, at present no resin systems have been obtained which have a satisfactory balance of curing ability, workability, adhesiveness, and connection reliability. Therefore, the need for anisotropic conductive adhesives which can connect at lower temperatures and in shorter times, as well as having excellent adhesiveness, connection reliability, storage stability, repairability, etc., has been strongly felt.

### Problems That the Invention Is to Solve

This invention was made as a result of various investigations concerning the problems of the prior art. Its purpose is to provide a thermosetting anisotropic conductive adhesive with excellent adhesiveness, connection reliability, storage stability, and repairability, which is able to form electrical connections between fine circuits, such as LCDs and TCPs or TCPs and PCBs, especially at low temperatures and in short periods of time.

### Means of Solving These Problems

The inventors discovered that, when connecting is performed by thermosetting, using an adhesive which consists of conductive particles dispersed in a composition which has both rapid curability at low temperatures and storage stability and which contains a radical-polymerizable resin, a radical-polymerizable monomer, an organic peroxide, a thermoplastic elastomer, a maleimide, and conductive particles dispersed in the resin composition, the cured radical-polymerizable resin and maleimide do not have sufficient electrical conductivity, curability, adhesiveness, workability, etc. As a result of investigating this problem, they discovered that a thermosetting anisotropic conductive adhesive which has excellent connecting properties can be obtained by adding a radical-polymerizable monomer to this adhesive. In this way, they arrived at this invention. That is, this invention is an anisotropic conductive adhesive which is characterized by the fact that it contains, as necessary ingredients, a radical-polymerizable resin (A), a radical-polymerizable monomer (B), an organic peroxide (C), a thermoplastic elastomer (D), maleimide (E), and conductive particles dispersed in these resin compositions, and the proportions of these ingredients, in parts by weight, are in the ranges of

- (1)  $(C)/\{(A)+(B)+(E)\}=(0.1-15)/100$
- (2)  $(B)/(A)=(1-90)/100$ .

### Working Embodiment of the Invention

The radical-polymerizable resins which can be used in this invention are not particularly limited; one can use resins which can be polymerized by radical-polymerization and which have one or more carbon-carbon double bonds in their molecules, e.g., vinyl ester resins, unsaturated polyester resins, diallyl phthalate, various acrylates, etc. Among these, vinyl ester resins are especially suitable because of their combination of curability with storability and the heat resistance, moisture resistance, and chemical resistance of the cured product. The term "vinyl ester resins" as used here refers to ones which are obtained by reacting epoxy resins with acrylic or methacrylic acid, or reacting glycidyl methacrylate and polyhydric alcohols. These resins may be used individually or in combinations of resins with different structures, molecular weights, etc. Moreover, in order to assure storability, one can also add polymerization inhibitors, such as quinones,

polyhydric phenols, phenols, etc., beforehand (e.g., Japan Public Patent Disclosure Bulletin No. 4-146951).

The radical-polymerizable monomers which can be used in this invention are not particularly limited; one can use ones which have both curability and flowability, e.g., 2-hydroxy-1,3-dimethanefuroxypropane [*sic*], dimethyl acrylamide, acryloyl morpholine, ethylene-glycol-modified bisphenol A diacrylate, ethylene-glycol-modified bisphenol F diacrylate, ethylene-glycol-modified paracumylphenol acrylate, N-vinyl-2-pyrrolidone, polypropylene glycol diacrylate, isocyanuric acid ethylene-glycol-modified diacrylate, isoamyl acrylate, lauryl acrylate, stearyl acrylate, butoxyethyl acrylate, phenoxyethyl acrylate, tetrahydrofurfuryl acrylate, isobornyl<sup>1</sup> acrylate, 2-hydroxyethyl acrylate, 2-hydroxypropyl acrylate, 2-hydroxy-3-phenoxypropyl acrylate, 2-acryloyloxyethyl succinic acid, 2-acryloyloxyethyl phthalic acid, 2-acryloyloxyethyl-2-hydroxyethyl phthalic acid, 2-acryloyloxyethyl acid phosphate, neopentylglycol diacrylate-1,6-hexanediol diacrylate, isooctyl acrylate, benzyl acrylate, nonylphenoxyethyl acrylate, ethylene-glycol-modified nonylphenol acrylate, 2-hydroxybutyl acrylate, perfluorooxylethyl acrylate, dimethyloltricyclodecane diacrylate, methyl methacrylate, ethyl methacrylate, n-butyl methacrylate, isobutyl methacrylate, 2-ethylhexyl methacrylate, isodecyl methacrylate, n-lauryl methacrylate, tridecyl methacrylate, n-stearyl methacrylate, cyclohexyl methacrylate, tetrahydrofurfuryl methacrylate, isobornyl<sup>1</sup> methacrylate, benzyl methacrylate, 2-hydroxyethyl methacrylate, 2-hydroxypropyl methacrylate, 2-hydroxyethyl acrylate, 2-hydroxypropyl acrylate, 2-hydroxybutyl methacrylate, dimethylaminoethyl methacrylate, diethylaminoethyl methacrylate, methacrylic acid, succinic acid, phthalic acid, glycidyl methacrylate, mono(2-methacryloyloxyethyl) acid phosphate, mono(2-acryloyloxyethyl) acid phosphate, ethylene glycol dimethacrylate, diethylene glycol dimethacrylate, 1,4-butanediol dimethacrylate, 1,6-hexanediol dimethacrylate, 5-butyl methacrylate, isostearyl methacrylate, behenyl methacrylate, n-butoxyethyl methacrylate, 2-phenoxyethyl methacrylate, 2-methacryloyloxyethyl-2-hydroxypropyl phthalate, glycerol monomethacrylate, 2-hydroxy-3-acryloyloxypropyl methacrylate, 3-chloro-2-hydroxypropyl methacrylate, 1,3-butanediol dimethacrylate, neopentylglycol dimethacrylate, 1,10-decanediol dimethacrylate, dibromoneopentylglycol dimethacrylate, dibromoneopentylglycol dimethacrylate, trifluoroethyl methacrylate, 2,2,3,3-tetrafluoropropyl methacrylate, 2,2,3,4,5,4-tetrafluoropropylbutyl methacrylate, perfluorooxylethyl methacrylate,  $\beta$ -methacryloyloxyethylhydrodiene phthalate,  $\beta$ -methacryloyloxyethylhydrodiene succinate, 3-chloro-2-hydroxypropyl methacrylate, stearyl methacrylate,  $\beta$ -acryloyloxyethylhydrodiene succinate, lauryl acrylate, etc. They may be used individually or in combination. Furthermore, by using radical-polymerizable monomers which combine the properties of strong adhesiveness to the objects being adhered, toughness of the cured product, etc., the anisotropic conductive adhesive can be designed in a simpler manner.

Furthermore, if the proportion of the radical-polymerizable monomer (B) to the radical-polymerizable resin (A) is less than  $(B)/(A)=1/100$ , the flowability and curability will be insufficient at the time of heating and adhering, and sufficient connection reliability cannot be obtained. If this ratio exceeds  $(B)/(A)=90/100$ , the workability will be reduced, and the adhesive force will be reduced due to excessive flowability during the heating and adhering.

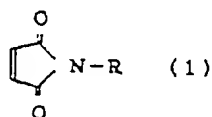
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1. Original: isoboniru

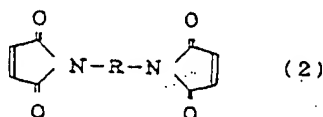
The organic peroxides used in this invention are not especially limited; for example, one can use 1,1,3,3-tetramethylbutylperoxy-2-ethylhexanoate, t-butylperoxy-2-ethylhexanoate, 5-hexylperoxy-2-ethyl hexanoate, 1,1-bis(t-hexylperoxy)-3,3,5-trimethylcyclohexane, 1,1-bis(t-hexylperoxy)-3,3,5-trimethylcyclohexane, bis(4-t-butylcyclohexyl) peroxydicarbonate, etc. These peroxides may be used individually, or they may be used as mixtures of 2 or more organic peroxides, in order to control the curability. Furthermore, it is also possible to add various polymerization inhibitors beforehand in order to improve the storage stability. Furthermore, they can also be diluted with solvents in order to facilitate the operation of dissolving them in the resins. Of course, the kinds of organic peroxides used in this invention are determined by the combinations of curability and storability of the adhesives achieved when the particular organic peroxides are compounded in them. Furthermore, if the ratio of the organic peroxide (C) to the radical polymerizable resin (A), the radical polymerizable monomer (B), and the maleimide (E) is less than  $(C)/\{(A)+(B)+(E)\}=0.1$  (in terms of parts by weight), the necessary thermosetting ability will not be obtained, and if this ratio is greater than  $(C)/\{(A)+(B)+(E)\}=15$ , the storage stability is decreased.

The thermoplastic elastomers used in this invention are not particularly limited; for example, one can use polyester, polyurethane, and polyimide resins, polybutadiene, polypropylene, styrene-butadiene-styrene copolymers, polyacetal resins, butyl rubber, chloroprene rubber, polyamide resin, acrylonitrile-butadiene copolymer, polyvinyl acetate resin, nylon, styrene-isoprene copolymer, polymethyl methacrylate resin, etc. Among these compounds, copolymers consisting primarily of acrylonitrile and butadiene are more advantageous for use, because they have excellent properties, including adhesiveness when used as anisotropic conductive adhesives, connection reliability, etc. It is also possible to use copolymers consisting primarily of acrylonitrile and butadiene to which various functional groups, such as carboxyl or hydroxyl groups, have been introduced.

The maleimides used in this invention are not particularly limited, as long as they are miscible with the radical polymerizable resins and thermoplastic elastomers, but in general, they are ones with the chemical structures shown in formulas (1) and (2). These chemical structures can of course be selected or modified according to the conditions of use of the anisotropic conductive adhesives (connection temperature and time) or the kinds of radical polymerizable [resin] and thermoplastic elastomer used.



(where R is a monovalent organic group)





(where R is a bivalent organic group)

The conductive particles used in this invention are not particularly limited, as long as they are conductive; one can use various kinds of metals or metal alloys, such as nickel, iron, copper, aluminum, tin, lead, chromium, cobalt, silver, gold, etc.; metal oxides; or particles of carbon, graphite, glass, ceramics, plastics, etc., which have surfaces coated with metals. The particle diameters of these conductive particles, their materials, and the quantities of them that are compounded can be selected as suitable, depending on the pitch and pattern of the circuit to be connected, the thickness and material of the circuit terminals, etc. Furthermore, suitable quantities of coupling agents may also be added to the anisotropic conductive adhesive of this invention, if desired. The purpose of adding coupling agents is to improve the adhesiveness of the anisotropic conductive adhesive at the adhesion interface and to improve the connection reliability by improving the adhesive strength, heat resistance, and moisture resistance. As the coupling agents, silane coupling agents are especially desirable, e.g., epoxy-silane, mercapto-silane, acrylic-silane (e.g.,  $\beta$ -(3,4-epoxycyclohexyl)ethyltrimethoxysilane,  $\gamma$ -glycidoxypropyltrimethoxysilane,  $\gamma$ -mercaptopropyltrimethoxysilane,  $\gamma$ -methacryloxypropyltrimethoxysilane, etc.).

According to this invention, the flowability during heating and adhering, the curability, the adhesiveness of the cured product, and its toughness are improved by the radical polymerizable monomer contained in an anisotropic conductive adhesive obtained by dispersing conductive particles in an adhesive consisting of a composition including a radical polymerizable resin, a radical polymerizable monomer, an organic peroxide, and a thermoplastic elastomer. Therefore, an anisotropic conductive adhesive is obtained which has excellent adhesiveness, connection reliability, storage stability, and repairability, and which can perform connections at extremely low temperatures and in extremely short times.

### Working Examples

This invention will be explained below by giving working examples and comparison examples.

#### 1. Preparation of adhesive resin composition

The materials shown in Table 1 were dissolved in MEK in the composition ratios of the non-volatile ingredients shown in Table 2 to obtain adhesive resin compositions.

#### 2. Preparation of anisotropic conductive adhesive

The compositions obtained according to 1. were poured out on 50  $\mu\text{m}$  polyethylene terephthalate films which had been mold-release-treated and drying was performed for 5 minutes in a 40°C oven to obtain anisotropic conductive adhesives in film form 15  $\mu\text{m}$  thick.

#### 3. Evaluation methods

The anisotropic conductive films obtained in the working and comparison examples were evaluated for their adhesive forces, connection reliabilities, and storabilities. The results are shown in Table 2. The objects which were adhered together using the adhesives were a TCP consisting of copper foil/polyimide=25/75  $\mu\text{m}$  with a 0.4  $\mu\text{m}$  tin plating (pitch 0.1 mm, 200 terminals) and a glass sheet 1.1 mm thick with an indium/tin

oxide film formed over its whole surface and a sheet resistance of  $30\ \Omega$  (below, "ITO glass").

Adhesive force: Measured by pressure-adhering at  $150^{\circ}\text{C}$ ,  $30\ \text{kg}/\text{cm}^2$ , and 15 sec and testing with a  $90^{\circ}$  peeling test.

Connection reliability: The connection resistance was measured immediately after the sample was prepared and after it had been left for 100 hours at a temperature of  $85^{\circ}\text{C}$  and a humidity of 85%. When the resistance could not be measured, the result was given as "OPEN."

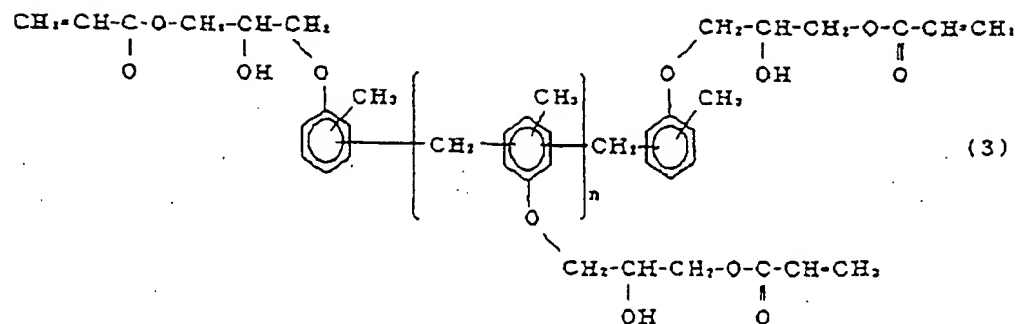
Storability: The connection resistance was measured after the anisotropic conductive film was left for 1 week at  $25^{\circ}\text{C}$ .

Table 1

Radical polymerizable resin	Novolak vinyl ester	Formula (3)
Radical polymerizable reactive monomers	(a) $\beta$ -Acryloyloxyethylhydrodiene succinate	Formula (4)
	(b) 2-Hydroxy-1,3-dimethacryloxypropane	Formula (5)
	(c) 2-Hydroxy-3-phenoxypropyl acrylate	Formula (6)
	(d) Acryloylmorpholine	Formula (7)
	(e) EO-modified paracumylphenol acrylate	Formula (8)
Organic peroxide	1-Hexylperoxy-2-ethyl hexanoate	
Thermoplastic elastomer	Acrylonitrile-butadiene-methacrylic acid copolymer	Formula (9)
Conductive particles	Ni/Au-plated polystyrene particles (mean particle diameter $5\ \mu\text{m}$ )	
Maleimide	Diaminodiphenylmethane-type bismaleimide	Formula (10)

【0022】

【化3】



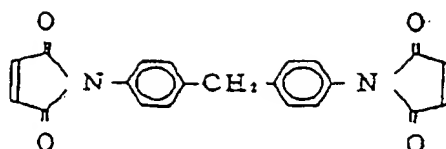
Carboxyl group quantity: 4.0 mol %

Acrylonitrile quantity: 27 wt %

Molecular weight: 100,000

[0029]

[化10]



(10)

Table 2

		Sym- bol	Working Examples					Comparison Examples					
			1	2	3	4	5	1	2	3	4	5	
Com- posi- tion	Radical polymerizable resin	[g]	(A)	150	150	150	150	150	150	150	150	150	150
	Radical polymerizable monomer (parts by weight)	(a)	37.5						225		37.5	37.5	
		(b)		37.5						225			
		(c)			37.5								
		(d)				37.5							
		(e)					37.5						
	Organic peroxide	(parts by weight)	(C)	25	25	25	25	25	25	25	0.3	70	
	Thermoplastic elastomer	(parts by weight)	(D)	300	300	300	300	300	300	300	300		
	Maleimide	(parts by weight)	(E)	200	200	200	200	200	200	200	200		
	Conductive particles	(vol %)	(F)	1	1	1	1	1 <sup>-</sup>	1	1	1	1	
Re- sults	Composition ratios: (C)/[(A)+(B)+(C)] (B)/(A)		6.5/ 100 25/ 100	6.5/ 100 25/ 100	6.5/ 100 25/ 100	6.5/ 100 25/ 100	6.5/ 100 25/ 100	6.5/ 100 0/ 100	6.5/ 100 150/ 100	0.08/ 100 25/ 100	18/ 100 25/ 100		
	Adhesive strength		[g/cm]	800	850	1400	1450	800	750	600	250	50	
	Connection reliability	Initial value After treatment	[Ω] [Ω]	1.1 1.2	1.2 1.5	1.2 1.6	1.4 1.8	1.2 1.6	Open Open	Open Open	Open Open	Open Open	
	Storability		[Ω]	1.5	1.5	1.7	1.8	1.4	Open	Open	Open	Open	

### Effects of Invention

The anisotropic conductive adhesive of this invention is able to make electrical connections between fine circuits, such as LCDs and TCPs, or TCPs and PCBs, especially at low temperatures and in short periods of time, and has excellent adhesiveness, connection reliability, storage stability, and repairability.